PATENT ABSTRACTS OF JAPAN

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(71)Applicant : TOYO KOHAN CO LTD

(72)Inventor : NOKITA NORIO
MIYAZAKI TAKAHIKO
INOUE NOBUO
FUJIZU SUSUMU

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(54) ALUMINA CERAMIC SINTERED COMPACT

(57)Abstract:

PURPOSE: To obtain an alumina ceramic sintered compact excellent in plasma ion etching resistance, etc., by heat-treating a sintered compact, comprising a high-purity Al2O3 and having a specified particle diameter and a specified density under conditions of a specified temperature and a specified time.

CONSTITUTION: This alumina ceramic sintered compact is obtained by forming an alumina powder, comprising [redacted].% aluminum oxide and the balance of an oxide of a metal (e.g. iron) other than aluminum and having about [redacted] average grain diameter, sintering the formed compact, preparing a sintered compact having [redacted] average grain diameter and 3[redacted]/cm3 density, then, as necessary, carrying out the grinding working of the resultant sintered compact and subsequently heat-treating the sintered compact at 1000-1550°C temperature for 0.1-6hr. The resultant alumina ceramic sintered compact is used for ultrahigh-density large scale integrated circuits and suitable for a part, etc., of a dry etching device for wafers and capable of improving the production yield of the wafers without falling off and scattering of ceramic particles from the surface even in undergoing the exposure to plasma ions.

* NOTICES *

CLAIMS

[Claim(s)]
[Claim 1] The alumina-ceramics sintered compact characterized by heat-treating the sintered compact whose densities 99.2 % of the weight or more and 99.99% of the weight or less of an aluminum oxide and the remainder consist of an oxide of metals other than aluminum, and mean particle diameters are 0.5 micrometers or more and 15 micrometers or less, and are three or more 3.88 g/cm, and three or less 3.97 g/cm, or the sintered compact which carried out the grinding process over 0.1 hours or more and 6 hours or less at the temperature of 1000 degrees C or more and 1550 degrees C or less.

[Translation done.]

* NOTICES *

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[The technical field to which invention belongs] this invention relates to a ceramic sintered compact. Still in detail, after sintering the Plastic solid of a ceramics, it is related with the alumina-ceramics sintered compact which is excellent in the dry etching-proof nature which performed heat treatment.
[0002]
[Description of the Prior Art] A ceramic sintered compact is built into the dry etching system using the plasma as a means which formation of a very detailed pattern replaces with the indispensable wet etching used [overly] in the manufacture field of a high-density large-scale integrated circuit using corrosion resistance, thermal resistance, an intensity, an electric insulation, and a property excellent in abrasion resistance. There are a chemical dry etching system which utilized the radical which occurs in (1) plasma as a dry etching system, reaction ion etching equipment using the rectilinear-propagation nature of (2) ion, an etching system using the plasma by (3) microwave magnetron discharge, etc. In order to generate the plasma of a high ion energy and high ion density, the temperature of an irradiation side becomes high and the impulse force by ion of especially an electronic cyclotron plasma etching system is also high.
[0003] Originally, although the ceramic sintered compact is excellent in an electric insulation and ion-proof spatter nature, when it is used as parts, such as a susceptor which supports the wafer of a dry etching system in fact, and a plasma ion irradiation is received, a ceramics evaporates from a sintering body surface, or ceramic grain carries out defluxion scattering of it by the impact of ion, and etching operation. Especially the ceramic grain that carried out defluxion scattering carried out the reattachment on the wafer, and had the problem reduce the production yield of a wafer.
[0004]
[Problem(s) to be Solved by the Invention] The alumina-ceramics sintered compact of this invention tends to prevent that alumina-ceramics grain carries out defluxion scattering from the base, when a plasma ion irradiation is received, and it tends to raise the production yield of a wafer. The manipulation width of face by etching of a wafer goes into a 0.5-micrometer age today, and it is predicted that it is set to 0.15 micrometers in the future. In this case, since setting to 0.3 micrometers or less the size of the grain which carries out defluxion scattering, and setting it 0.07 micrometers or less in the future corresponds to the demand of such an age demanded, even if it receives the etching operation by plasma ion, it is necessary to develop the ceramic sintered compact without defluxion scattering of ceramic grain.
[0005]
[Means for Solving the Problem] Then, after sintering the alumina-ceramics Plastic solid fabricated in the desired configuration the place which was inquiring that the ceramic sintered compact without defluxion scattering of ceramic grain should be developed, even if it receives irradiation of plasma ion in a vacuum or under 0.01-5Pa etching gas presence by performing afterbaking processing which carried out the grinding process of the sintered compact further, it finds out that it is possible to prevent

defluxion scattering of alumina-ceramics grain. That is, the alumina-ceramics sintered compact of this invention is characterized by heat-treating the sintered compact whose densities 99.2 % of the weight or more and 99.99% of the weight or less of an aluminum oxide and the remainder consist of an oxide of metals other than aluminum, and mean particle diameters are 0.5 micrometers or more and 15 micrometers or less, and are three or more 3.88 g/cm, and three or less 3.97 g/cm, or the sintered compact which carried out the grinding process over 0.1 hours or more and 6 hours or less at the temperature of 1000 degrees C or more and 1550 degrees C or less.

[0006]

[Embodiments of the Invention] A ceramic sintered compact can be manufactured by any technique of an ordinary-pressure sintering process, a hot pressing, or a hot isostatic pressing. A ceramic Plastic solid passes through the process which was solidified simultaneously with heating or heating and which is back-cooled in such technique. In case the ceramic sintered compact solidified especially at the elevated temperature is cooled, since the temperature of a core part is high, compared with the surface section of a ceramic sintered compact, tensile stress occurs on a front face. When tensile stress exceeds the tensile strength in the temperature, a micro crack occurs in a grain boundary. Thus, since the point of a micro crack which occurred is an acute angle extremely, the crack sensitivity is also high.

[0007] When plasma ion is irradiated at the ceramic sintered compact in such status, since [on the front face of ceramic] a surface layer is heated very much, opening of a crack is closed and compressive stress occurs. If this heating and cooling are repeated, ****-compression will be repeated, a micro crack will develop gradually, and finally it will be started by striking it plasma ion, and it will disperse as ceramic grain. Even if the point of a micro crack came to be roundish, the crack sensitivity fell and it received irradiation of plasma ion by performing afterbaking processing even if it is such a ceramic sintered compact, it found out that it was possible to inhibit scattering of ceramic grain.

[0008] Furthermore, a ceramic sintered compact is used in many cases, after giving a grinding process using a diamond wheel etc. In this case, although a crack is produced in a grinding-process side, there are two kinds of cracks, the crack produced by heating by manipulation and cooling by the grinding fluid and the crack produced by grinding-process asymmetry. Although the number of books per unit area of these cracks and the length of the crack to the depth orientation from a surface layer are different with a material property and grinding-process conditions, they also attain to some dozens of micrometers. The point of a crack which occurred with the stress at the time of a grinding process is an acute angle, and its crack sensitivity is also high. If plasma ion is irradiated especially in the grinding-process side of a ceramic sintered compact, a skin temperature will rise, there has also been the length of a crack for a long time, and it is more remarkable than the case where extension of a crack is the sintered compact with which a grinding process is not given. Consequently, it is in more inclinations than the case where the amount which strikes to the plasma ion of ceramic grain, is taken out, and disperses has not carried out a grinding process.

[0009] Even if it is the ceramic sintered compact which has a grinding-process side, by performing afterbaking processing, grinding-process asymmetry disappears mostly. Even if the point of a micro crack came to be roundish again, the crack sensitivity fell and it received irradiation of plasma ion, it became clear that it is possible to inhibit scattering of ceramic grain. Furthermore, it also became clear the grinding waste adhering to the grinding side which was not able to be removed by ultrasonic cleaning, either, and that it becomes possible to make a grinding side fix by performing afterbaking processing.

[0010] Since there are few amounts which evaporate even if especially an aluminum-oxide (henceforth alumina) machine sintered compact has the low vapor pressure of an alumina and a ceramics receives irradiation of plasma ion and it excels in dry etching-proof nature when manufacturing a ceramic sintered compact by the technique described above, when it uses as a material of the parts made from a ceramics for dry etching systems, a desirable result is obtained especially. this invention is explained, using as an example the case where the ceramic parts for dry etching are manufactured below using an alumina machine sintered compact.

[0011] Having made into 99.2 % of the weight or more purity of the alumina used for the parts made

from a ceramics of this invention is based on the ground described below. That is, although the oxide of metals other than the aluminum of the remainder exists in the type which covers an alumina grain to the grain boundary of an alumina as a multiple oxide, if these multiple oxides have high vapor pressure and irradiation of plasma ion is received compared with an alumina, they will evaporate, and produce the status that the alumina grain was isolated. The particle diameter of an alumina is large, and the thickness or grain-boundary layer thickness of a multiple oxide which has covered the alumina grain becomes thick so that there are many amounts of oxides other than an alumina, and vaporization of a grain-boundary fraction is remarkable.

[0012] When the purity of an alumina is [a particle diameter] 15 micrometers or more at 99.2 or less % of the weight, even if the thickness or grain-boundary layer thickness of a multiple oxide is thick and performs afterbaking processing, it stops the defluxion scattering prevention effect of alumina grain accepting. Therefore, purity of an alumina is set and a particle diameter is set to 15 micrometers or less 99.2% of the weight or more. In case an alumina sintered compact is manufactured, the alumina particle diameter of available raw material powder is 0.1 micrometers or less, and the lower limit of a particle diameter was set to 0.5 micrometers because it grew up to 0.5 micrometers by the sintered-compact manufacturing process. In addition, in order that the amount of the multiple oxide with the high vapor pressure which exists in a grain boundary may decrease as the alumina with high purity, the alumina sintered compact stabilized more is obtained. The purity of the desirable alumina in that case is 99.5 % of the weight or more and 99.99% of the weight or less of a domain. Moreover, the particle diameters which are easy to manufacture what has the fine particle diameter of an alumina as an alumina sintered compact although the thickness of a grain boundary becomes thin are 2 micrometers or more and 6 micrometers or less. Therefore, as for the particle diameter of an alumina, it is more desirable that they are 2 micrometers or more and 6 micrometers or less.

[0013] The example of the manufacture technique of the alumina sintered compact by the ordinary-pressure sintering process using the above-mentioned alumina powder is described below. After the aluminum oxide of 99.9 % of the weight of purity and the remainder consist of ~~metallic oxides, such as sodium, calcium, magnesium, silicon, and iron~~, make water a solvent using the alumina powder of 0.1-10 micrometers of mean particle diameters and carry out little addition of a polycarboxylic acid, stearin acid, and the polyvinyl alcohol as a binder, trituration mixture is carried out with a pot mill. After considering as the granulatio which dries the slurry which carries out trituration mixture and is obtained by the spray dryer, and is rich in a fluidity, a mechanical press or a hydrostatic-pressure press is used, and it is 2 0.8-1.5t/cm. A pressing is carried out. Next, a green compact is calcinated at 1450-1650 degrees C in the atmospheric air for 0.5 to 6 hours, and turns into a sintered compact. The optimum conditions of a compacting pressure, burning temperature, and a firing time change with characters of the raw material to use.

[0014] Moreover, the particle diameter and density of a sintered compact also change with characters of a raw material. The density of a sintered compact is set to three or more 3.88 g/cm, and three or less 3.97 g/cm for a mechanical strength being low in three or less 3.88 g/cm, and an upper limit is because a three or more cm [3.97 g/cm] thing is not obtained. Next, a grinding process may be given, in order to make a sintered compact into the dimension of the last product configuration, and in order to make a front face into a smooth field. As for a grinding process, it is desirable to carry out using a diamond wheel etc., so that the granularity after a manipulation may be set to 15 micrometers by the maximum height (difference of the summit of Rmax:split face and a bottom).

[0015] It is indispensable to perform afterbaking processing to a sintered compact or the sintered compact with which the grinding process was given finally in this invention. An afterbaking is carried out over 0.1 hours or more and 6 hours or less at the temperature of 1000 degrees C or more and 1550 degrees C or less. The ground for having made heating temperature into 1000 degrees C or more and 1550 degrees C or less is for producing the thing which the creep by the heat of a sintered compact will become remarkable if elimination of the asymmetry which remains in grain, an improvement of the crack sensitivity of a crack fraction, and elimination of the multiple oxide which exists in a grain boundary are inadequate below 1000 degrees C and it heats at 1550 degrees C or more, and deformation

becomes large, and big and rough-ization of crystal grain.

[0016] The ground for having made the heating time of afterbaking processing into 0.1 hours or more and 6 hours or less is stopping an improvement of an effect accepting, even if elimination of the asymmetry which remains in grain, an improvement of the crack sensitivity of a crack fraction, and elimination of the multiple oxide which exists in a grain-boundary fraction are inadequate in 0.1 or less hours and it heats 6 hours or more. In addition, unless a ceramics deteriorates, even if it carries out afterbaking processing in a vacuum, the atmospheric air, and which [of specific gas] the ambient atmosphere, it does not interfere.

[0017] By observing by the scanning electron microscope, the effect of heat-treatment can be checked very easily after this. That is, it is observed by giving an afterbaking that a grain-boundary layer appears on the front face of a sintered compact or the front face which performed the cutting after sintering. Moreover, the crack produced at the time of a grinding process can also check disappearing by the afterbaking easily by observation by the scanning electron microscope.

[0018] The ceramic sintered compact described above is not limited only to an application on the parts for dry etching systems, and can be applied also to the ceramic parts which are used under the environment where alkali, an acid, or the acidic solution containing especially fluoric acid exists and with which high corrosion resistance is demanded.

[0019]

[Example] Hereafter, based on an example, this invention is explained still in detail.

(Example 1) Purity:99.2% of the weight of an alumina sintered compact, diameter:of mean crystal grain12.0micrometer, and density:3.89g/cm3 By the atmospheric pressure method, the sintered compact was manufactured as ceramic parts of the dry etching system for 6 inch wafers using an electronic cyclotron plasma. This part consists of three points of the ring placed on the susceptor which supports covering and the wafer of an electrode, the wait which presses down a wafer, and a wait. The grinding process was given to things other than a ring among the above-mentioned component parts of three points, it finished in the predetermined dimension, and the afterbaking of each component part of three points was carried out at 1500 degrees C in the atmospheric air for 1 hour. In addition, the same parts as an example 1 were also manufactured except not carrying out an afterbaking for the comparison. As a result of equipping the above-mentioned dry etching system for 6 inch wafers with these parts and actually carrying out etching of a wafer, 3 or that five pieces have adhered accepted the grain which has the diameter 0.3 micrometers or more generated from the parts of this invention on the 6 inch wafer. On the other hand, when the comparator article which does not carry out an afterbaking was used, 25 or 42 adhesion accepted. That is, it became clear that adhesion of a up to [the 6 inch wafer of the grain which has a diameter 0.3 micrometers or more] decreases to conventional one eighth by using the ceramic parts of this invention.

[0020] (Example 2) Purity:99.8% of the weight of an alumina sintered compact, diameter:of mean crystal grain4.0micrometer, and density:3.92g/cm3 The same sintered compact as an example 1 was manufactured as the same ceramic parts as an example 1 except being a sintered compact. Furthermore, what carried out the same afterbaking as an example 1 on these ceramic parts, and the thing which does not carry out an afterbaking for a comparison were prepared, and etching same in an example 1 was carried out. Consequently, the number of the grain which has the diameter 0.3 micrometers or more which adheres on a wafer when an afterbaking is carried out was [20 or] 35 when 0 or one piece, and an afterbaking were not carried out.

[0021] (Example 3) After giving an afterbaking to the alumina sintered compact of the example 1 and the example 2 at various time and temperature, the sintered compact was etched by argon ion by 200V-10W in the 3.0Pa argon ambient atmosphere for 3 hours using planar magnetron spatter equipment, etching loss in quantity was measured, and the result was shown in Table 1. Even when it excels in etching-proof nature and the sintered compact with low purity is used so that the purity of a sintered compact was high and afterbaking temperature was high, it is possible to improve etching-proof nature by carrying out afterbaking processing by high temperature.

[0022]

[Table 1]

（表１）　後加熱後のエッチング減量

		各加熱時間後のエッチング減量　(mg)			
加熱温度(°C)	加熱時間(hr)	0.1	1	3	6
1000	実施例１	74	70	63	60
	実施例２	48	48	47	47
1250	実施例１	67	64	58	55
	実施例２	48	47	47	46
1550	実施例１	59	55	52	52
	実施例２	46	43	40	39
後加熱なし	実施例１	76			
	実施例２	48			

[0023]
[Effect of the Invention] The alumina-ceramics sintered compact of this invention is excellent in plasma-proof ion etching nature, and defluxion scattering of the ceramic grain from the ceramic front face at the time of receiving irradiation of plasma ion is inhibited. Therefore, the production yield of a wafer can be raised.

[Translation done.]